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Pilot Scale Study on Salt Removal by Electrodialysis from RO Concentrate with High Scaling Potential

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Abstract
This paper presents a pilot scale investigation for salt removal by electrodialysis on the Reverse Osmosis (RO) concentrate of the IWVA that is obtained by RO treatment of the effluent of a WWTP. Due to the presence of a high concentration of carbonates (around 1300-1600 ppm as carbonates) the RO concentrate has a high scaling potential and a decarbonation process based on an acidification with HCl and an aeration was installed. In this study, it is proved that the ED system can run a long-term operation on the decarbonated RO-concentrate to produce the designed effluent (around 75% salt removal) without scaling problems. Meanwhile, a high overall water recovery (95%) was achieved for the integrated RO-ED system. Furthermore an economical analysis of the ED system was carried out to evaluate the feasibility of this process. Result shows that the full operational cost is around 0.66 Euro/m^3 RO-concentrate treated by the ED system; the total cost of the RO-filtrate is thus enhanced with 0.22 Euro/m^3.

Keywords: electrodialysis, RO concentrate, salt removal, wastewater reclamation

Introduction
Nowadays, water shortage has become a global issue (IWMI, 2006). Water reuse, wastewater reclamation and desalination of saline water are crucial strategies to deal with the global water need aimed at economical development (Miller, 2006; OECD, 2009; Dolnicar and Schäfer, 2009). It is recognized that water supply and sanitation in developing countries, in view of the Millenium Development Goals (MDG) is even more important (Palaniappan, 2009). Thus, water and wastewater treatment technologies are being developed and applied to fulfill the higher requirements on water quantity and quality. As one of the fastest growth water treatment processes since the end of the 20th century, reverse osmosis (RO) is getting recognition from various successful applications (Drioli and Fontananova, 2004; Pearce, 2007) due to the high and stable quality of the water produced, relatively low cost and less chemical adding.

However, there are still some drawbacks for RO application. One of the most important issues is the treatment of the concentrated waste drain from RO due to the high salinity of this RO concentrate. Traditionally, the RO concentrate is discharged into the natural water body (with or without dilution, depending on the local discharge regulations to avoid degradation of the water body) or treated by evaporation process. The former method is not environmental friendly and the latter process is very costly.

Reahl (1990) reported the design and the operation of an UF-RO-EDR system to reclaim the wastewater from an electronic and aerospace facility in the southwestern United States. The project aimed at reducing the volume of the RO concentrate by using the electrodialysis reversal process (EDR):85% of the RO concentrate could be reinserted to the UF-RO system and the overall water recovery improved to 97%. The system was installed in late 1985 and was successfully operated over 12,000 hours (Reahl, 1990). Similarly, an RO-EDR system was applied to treat the brackish water with high scaling potential from Sahel region of Tunisia and the system overall water recovery reached 91.6% (Turek et al., 2009). Korngold et al. (2005, 2009) investigated the treatment of the concentrate from an RO unit fed by brackish water by a pilot ED installation with a separated gypsum precipitator to reduce the scaling problem during the ED operation. The salts in the RO concentrate were partially removed by the ED and the produced water was mixed together with the RO permeate. The overall recovery of the RO unit was improved and furthermore, the volume of brine in the combined process was reduced, at the same time increasing its salt concentration. The salt concentration in the brine increased from 1.5% to 10% and the RO unit overall recovery increased to 97%-98% (Korngold et al., 2009).

In conclusion from these studies, ED (or EDR) shows remarkable advantages technically and economically on treating RO concentrate streams. Although some investigations on the treatment of RO concentrate by ED process have been done, further study on the details of the process is needed, in view of fully understanding the influence of operational parameters and transport of the inorganic and the organic compounds through the membranes. Furthermore, scaling on the membranes and fouling remain potential hurdles.

In July 2002 the Intermunicipal Water Company of the Veurne region (I.W.V.A., Wulpen, Belgium) has started to treat wastewater for producing infiltration water for groundwater recharge of the dune water catchment 'St-André'. As this water is recharged in a dune area, which is of high ecological value, the infiltration water must have low levels of salts and nutrients. Based upon the quality parameters set for the infiltration water, a combination of membrane filtration (UF and RO) was chosen to treat the wastewater (mainly domestic) effluent.

The schematic diagram of the wastewater treatment process is shown in Figure 1.The intake of the wastewater first passes a mechanical screen with 1 mm openings to remove all bigger particles. Then after the conventional biological (anaerobic-aerobic) treatment, the water is clarified and flows to the submerged hollow fiber UF units. There are 5 UF units, each unit contains 3120 m^2 ZeeWeed® membranes (Zenon, Canada), which have a maximum pore size of 0.1 µm with the outside-in flow. The UF units can treat a maximum of 450 m^3h^{-1} of effluent with a minimal recovery of 85 %. From the UF filtrate reservoir the water first passes the cartridge safety filters with pore sizes of 15 µm, and is then pumped to the RO system. The RO system contains 36 pressure vessels with the brackish water RO membrane elements (8-inch BW 30LE-440 DOW) in a two-stage configuration. The system totally has 15744 m^2 of membrane area and can treat a maximum of 410 m^3h^{-1} of UF filtrate. The recovery of the RO system is around 75 % and is varied according to feed water conductivity. Finally, the RO product (permeate) is pumped for infiltration; and the RO brine (concentrate) is mixed with the UF concentrate and is discharged to the canal.

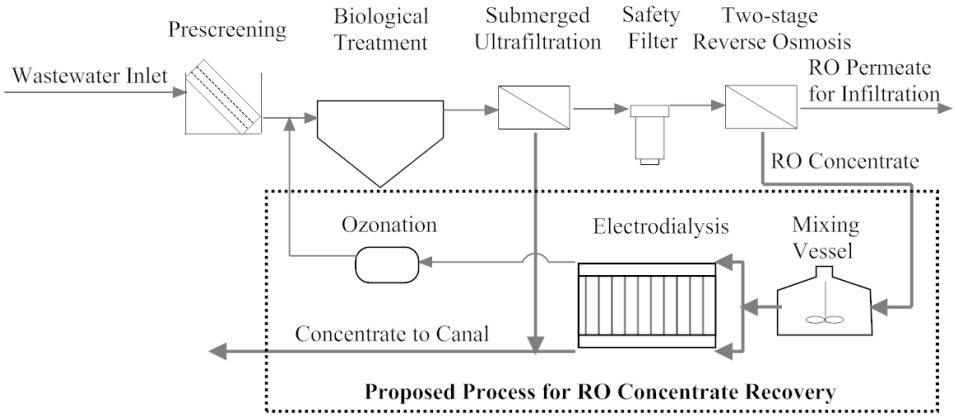

Figure 1 Schematic diagram of I.W.V.A wastewater treatment process

This work presents a treatment process by a pilot scale ED installation of the RO concentrate with high scaling potential, to improve the global water recovery of the wastewater treatment plant (WWTP). Decarbonation by hydrochloric acid on the ED concentrate has been done to exam the efficiency on reducing the scaling potential of the ED concentrate. During the experiments, the transport mechanisms of the inorganic ions and the organic compounds were studied. The system was examined under stable operational conditions and the results show that the global water recovery can be increased from around 75% to 95. Furthermore, an economical analysis is carried out to evaluate the cost effectiveness of the ED pilot system.

Materials and Methods

Membrane and the installation

A PCCell ED 1000H (PCCell GmbH, Heusweiler, Germany) electrodialysis stack was installed in the pilot scale skid (IEC NV, Riemst, Belgium). The set-up consists of three separated circuits with three 200-Litre vessels for the diluate, the concentrate and the electrolyte rinsing solution. For each membrane, the active surface area is 0.1 m^2 and the flow channel width between two membranes is 0.5 mm. There are 25 cell pairs in the stack, each containing a diluate and a concentrate compartment, so in total 25 pieces of anion exchange membranes and 26 pieces of cation exchange membranes are used. Thus, the total surface membrane area in the stack is 5 m^2 (2 x 2.5 m^2). PC-SK standard cation exchange membranes and PC-SA standard anion exchange membranes are used in the stack. Information about the membranes is given in Table 1, which was supplied by the manufacturer (PCA- Polymerchemie Altmeier GmbH, Heusweiler, Germany).

Table 1 Characteristics of the PCA standard cation and anion exchange membrane

Membrane	Thickness (μm)	Ion exchange capacity (meq/g)	Chemical stability (pH)	Permselectivity	Functional groups	Surface Potential ($\Omega.cm^2$)
PC-SK	130	ca. 1	0-11	>0.96	$-SO_3Na$	0.75-3
PC-SA	90-130	ca.1.5	0-9	>0.93	$-NR_4Cl$	1-1.5

During the experiments, the RO concentrate was used for both the diluate and the concentrate streams in the ED. Initially the feed of the ED-system (RO concentrate) was decarbonated, later only the ED concentrate was decarbonated. The decarbonation process will be described in Results and discussion part of this paper. Due to rainfall and seasonal change, the conductivity of the decarbonated RO concentrate varies between 3.0-5.8 mS/cm. The electrolyte

rinsing solution was H_2SO_4 0.1M. The pilot scale experiments were conducted by two different operational methods: batch mode and feed-and-bleed mode.

1) Batch mode

During batch mode, a constant current was applied; the diluate, the concentrate and the electrolyte rinsing solution were recirculated in the system until the salt removal reached around 75%. Based on optimization of the operational parameters, the current was set as 5 A, which corresponded with a current density 50 $A.m^{-2}$; and the flow rate was set as 500 $L.h^{-1}$ for all of the circulation streams. The batch mode was applied to reduce the salt concentration to the target (75% of removal) prior to the feed-and-bleed mode.

2) Feed-and-bleed mode

During the feed-and-bleed mode, the applied current dropped to 2.6 A; the circulation flow rates of the three circuits were the same as applied in the batch mode. However, 75 $L.h^{-1}$ of the effluent of the stack was withdrawn from the system. This stream was regarded as the product of the ED system which in practice would be reinserted into the biological treatment unit of the WWTP. Under the optimized condition, it should contain around 25% of salts compared with the feed (the decarbonated RO concentrate). By using this procedure, the global water recovery of the whole system can be increased from around 75% to 95% percent (Figure 2).

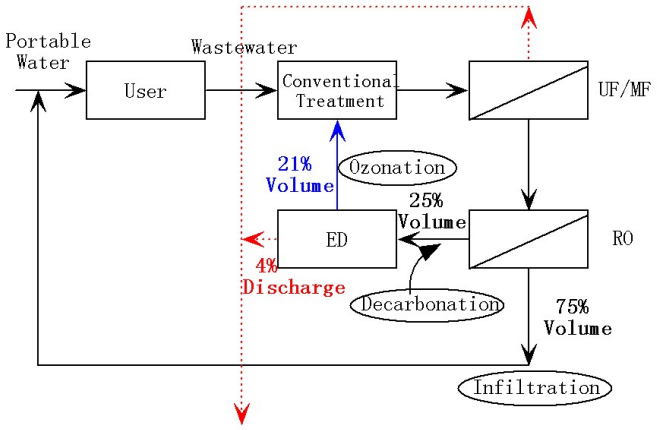

Figure 2 The proposed procedure and the water volume (by percentage) balance in the proposed system

Analytical methods

All the chemicals in the experiments were analytical grade. Samples were collected every 30 minutes. Anion and cation concentrations were measured by Ion Chromatography (DIONEX series 4500i with IONPAC AS14 Analytical Column for anion analysis and DIONEX DX-100 with IONPAC CS14 for cation analysis, DIONEX, USA). The eluent for anion analysis contained 3.5 mmol L^{-1} CO_3^{2-} and 1.0 mmol L^{-1} HCO_3^-; and the eluent for cation analysis contained 10 mmol L^{-1} CH_3SO_2OH. The concentration of bicarbonate and organic compounds were measured as Total Inorganic Carbon (TIC) and Total Organic Carbon (TOC) using a TOC Analyzer (TOC-V CPN E200V TOC Analyzer, Shimadzu, Japan).

Data analysis

Selectivity, represented as separation efficiency, was calculated by the method introduced in literature by Van der Bruggen et al. (2004). In this method, the separation efficiency S between component A and B is evaluated as:

$$S(t) = \frac{(c_A(t)/c_A(0)) - (c_B(t)/c_B(0))}{(1 - c_A(t)/c_A(0)) + (1 - (c_B(t)/c_B(0))} \times 100\%$$

The calcium sulphate saturation level was calculated by Debye-Hückel Theory (Debye and Hückel, 1923). At pH 6.5-9.5, the Langelier Saturation Index (LSI) is used to indicate the scaling potential of calcium carbonates containing water. LSI value was calculated by the method below:

$LSI = pH - pH_S$

where, pH_S is the "saturation pH" in which the water is saturated with calcium carbonates, pH_S is defined as: $pH_S = (9.3 + A + B) - (C + D)$

where,

$A = (log_{10}[TDS] - 1)/10$, [TDS] is the concentration of the total dissolved solid (mg L^{-1})

$B = -13.12 \times \log_{10}(K) + 34.55$, K is the Kelvin Temperature;

$C = \log_{10}[C^*_{Ca^{2+}}] - 0.4$, $[C^*_{Ca^{2+}}]$ is the concentration of Ca^{2+} as $CaCO_3$ (mg L^{-1})

$D = \log_{10}[Alk^*]$, $[Alk^*]$ is the concentration of alkalinity as $CaCO_3$ (mg L^{-1})

Results and discussion

Decarbonation process

Due to the presence of a high concentration of carbonates (around 1300-1600 ppm as carbonates), there is a high scaling potential when running the ED system. Table 2 shows the concentration of the main ions in the typical feed water. Results indicate that the ions that cause the scaling potential are Ca^{2+}, Mg^{2+} and CO_3^{2-}, due to the LSI of the feed water reaching 2.21, which means the scaling potential of Ca^{2+} and Mg^{2+} carbonates is high and if concentrated 10 times, the LSI will reach 4.11. However, the scaling potential of SO_4^{2-} is relatively low in the feed water, the saturation level δ is only 5.4%; even concentrated 10 times, it just reaches saturation (105.1%). Thus, carbonate is the main matter which could cause scaling.

Hence, decarbonation is essential for the ED system operation stably and safely. The decarbonation process in this work used continuous dosing of HCl (28% weight concentration) under aeration The pH was adjusted to 5 ± 0.5. The principle is described as the equilibrium below:

$$CO_3^{2-} + H^+ \Leftrightarrow HCO_3^- \qquad HCO_3^- + H^+ \Leftrightarrow H_2CO_3 \qquad H_2CO_3 \Leftrightarrow H_2O + CO_2(l)$$

$$CO_2(l) \Rightarrow CO_2(g)$$

Table 2 Typical feed water and the simulated concentrated water (×10) ions content and the scaling potential evaluation at pH 7.8, 25°C

	Ion concentration (mg L^{-1})								Scaling Potential	
	Na^+	K^+	Ca^{2+}	Mg^{2+}	Cl^-	NO_3^-	HCO_3^-	SO_4^{2-}	δ, %	LSI
Feed	722	154	426	49	974	113	1377	433	5.4	**2.21**
Simulated concentrate	7220	1540	4260	490	9740	1130	13770	4330	**105.1**	**4.11**

Pilot scale ED operation

Batch experiment

Based on the optimized operational parameters, a current of 5 A and a flow rate of 500 L h^{-1} for all the streams (diluate, concentrate and electrolyte rising solution) were applied for the

batch experiment on the pilot-scale installation.

The results show that the decarbonation process sufficiently reduced scaling potential of the feed and (the initial concentrate of ED). After this decarbonation it was possible to operate the ED-installation with a satisfying salt removal and this without losing much of the TOC. The results after 2 hours of desalination are shown in Table 3. A slight selectivity has been found between the removal of monovalent and multivalent cation and anions using non-selective anion- and cation- exchange membranes (Table 4), which corresponding well with the previous investigation (Zhang et al., 2009).

Table 3 Ions and organic compounds removal efficiency by pilot ED installation (Batch mode with decarbonated RO concentrate)

	HCO_3^-	Cl^-	NO_3^-	SO_4^{2-}	Na^+	K^+	Mg^{2+}	Ca^{2+}	TOC
Feed water	28	758	56	401	662	116	39	353	31
Mean value	16	128	6	129	108	10	3	30	29
Removal efficiency (%)	42	83	89	68	84	92	92	92	6

Table 4 Selectivity of SK (standard cation exchange membrane) and SA (standard anion exchange membrane) in pilot experiment

Selectivity	120 min
Cl^-/SO_4^{2-}	0.23
Cl^-/NO_3^-	-0.02
Na^+/K^+	-0.13
Na^+/Ca^{2+}	-0.15
Na^+/Mg^{2+}	-0.19

Feed-and-bleed experiment

A batch experiment was done to decrease the conductivity in the diluate vessel to the target concentration (around 25% of the initial conductivity) prior to shift the system to the feed-and-bleed mode. NaCl solution with a conductivity of 5.5 mS cm^{-1} was used as initial ED-concentrate and there was no discharge of the concentrate during the experiment. A short-term feed-and-bleed experiment (300 min, 75 $L.h^{-1}$) was carried out to examine the system stability. No scaling was observed in the experiment and the system showed a good stability.

After an initial batch step a long-term feed-and-bleed experiment (75 $L.h^{-1}$) was performed on the pilot installation. It should be noted that the conductivity of the feed (decarbonated RO concentrate) was 3.5 mS cm^{-1} in the first 20 hours and 2.8 mS cm^{-1} between 20 and 42 hours; this phenomenon was due to heavy rainfall. During the experiment, the voltage was stable (15 V in period 1 and 25 V in period 2) and the concentrate flow pressure only changed in the range of 5%. In period 1 the conductivity dropped 69 %, in period 2, 79 %. The electric power (kW) is calculated by multiplying the current (2.6 A) and the voltage and it represents the consumed electricity power by the stack. Currently in I.W.V.A., 1 kWh costs 9 eurocents (€c), which is used for the economical evaluation**.** A cost of 0.35 and of 0.58 eurocents was calculated for power consumption by the stack during 1 hour of operation in period 1 and 2 respectively. From this data a cost of 4.7 and 7.7 eurocents was calculated for power consumption of the stack for the treatment of 1 m^3 of decarbonated RO concentrate under the conditions applied.

Further economical analysis

The effects of current and flow rate were evaluated operating the pilot instrument in the continuous mode and the results were used for an economical analysis. For the current effect, 6 different currents with a constant flow rate of 300 $l.h^{-1}$ were used; for the flow rate effect, 5 different flow rates with a constant current of 5A were used. The cost of pumping is based on the energy consumption of the 3 pumps: 8.18 kWh/day in the current pilot installation. The cost of pumping in Table 5 is however for "optimized pumps", which means that we only consider the cost for the feed of the stack and not that of the excess capacity of the pumps that is circulated back to the vessels.

The intrinsic removal capacity is calculated by multiplying the salt removal (%) by the applied flow rate. To obtain the specific cost (energy), the electricity cost of the stack and the pumping are summed up and this value is divided by the intrinsic removal capacity ($\%.m^3/h$). This specific cost (energy) indicates how much it costs to remove 1% of the salts from 1 m^3 of the feed water by the pilot system. Thus, the cost to produce the target stream (75% of salt removal) is calculated by multiplying the "specific cost" with 75, which is shown in the column "cost for 75% of removal". However, the cost of decarbonation (aeration and adding of HCl to the RO concentrate) should also be taken into account: this was calculated as 9 eurocents for the aeration and 41 eurocents for the acid (0.14 Euro. L^{-1}) and is thus very important. The "Total cost" for the pilot ED system to produce 1 m^3 of diluate can be calculated by adding "cost for 75% of removal (energy)" and "cost for aeration and acid". The analysis shows that in the flow rate range of 200-500 L/h the lower the flow rate, the lower the cost to produce $1m^3$ of product.

Table 5 Cost analyses of different flow rates with a constant current of 5A

Flow rate	Electric power	Cost	Cost of pumping	Intrinsic removal capacity	Specific cost (Energy)	Cost for 75% of removal (Energy)	Cost for aeration and acid (HCl)	Total cost
(L/h)	(kWh/h)	(€c/h)	(€c/h)	($\%.m^3/h$)	(€c/$\%.m^3$)	(€c/m^3)	(€c/m^3)	(€c/m^3)
500	0.053	0.477	3.068	8.400	0.422	31.647	50	**82**
400	0.051	0.459	2.454	8.680	0.336	25.170	50	**75**
300	0.052	0.468	1.841	8.880	0.260	19.497	50	**69**
250	0.053	0.473	1.534	8.700	0.231	17.295	50	**67**
200	0.055	0.495	1.227	9.180	0.188	14.069	50	**64**

Table 6 Cost analyses of different current with a constant flow rate of 300 L/h

Current	Electric power	Cost	Cost of pumping	Intrinsic removal capacity	Specific cost (Energy)	Cost for 75% removal (Energy)	Cost for aeration and acid (HCl)	Total cost
(A)	(kWh/h)	(€c/h)	(€c/h)	($\%.m^3/h$)	(€c/$\%.m^3$)	(€c/m^3)	(€c/m^3)	(€c/m^3)
1	0.003	0.027	1.841	1.590	1.175	88.090	50	**138**
3	0.023	0.211	1.841	5.490	0.374	28.020	50	**78**
5	0.057	0.509	1.841	8.700	0.270	20.250	50	**70**
7	0.103	0.926	1.841	11.880	0.233	17.466	50	**67**
9	0.171	1.539	1.841	15.990	0.211	15.851	50	**66**
11	0.307	2.762	1.841	20.010	0.230	17.251	50	**67**

Similarly, the economical analysis of different currents with a constant flow rate of 300 l/h can be obtained, the results are shown in Table 6 above. It reveals that the system is the most cost-effective at a current of around 7-9A at 300 L/h of flow rate.
In this type of process the main costs are thus due to the pretreatment of RO-concentrate (50 eurocents) which is necessary to avoid scaling during operation. In the future we will try to decrease acid consumption by applying the decarbonation process to the ED concentrate. The operational costs are mainly due to the pumping costs. The stack it selves is responsible for only a small amount of the total costs. In the best combinations the total cost is about 66 eurocents per m^3 of treated RO-concentrate.
The RO-concentrate is obtained as a by stream of the preparation of the RO-filtrate in a relative amount of 1 to 3. The cost for the preparation of 3 m^3 of RO-filtrate is thus enhanced with 66 eurocents and thus the cost for the preparation of 1 m^3 RO water is augmented with 22 eurocents.

Conclusion

The decarbonation process sufficiently reduced scaling potential of the feed and the initial concentrate of ED. After this decarbonation it was possible to operate the ED-installation under steady-state long-term experiment (42 hours) condition with satisfying salt removal. A slight selectivity was observed between the removal of monovalent and multivalent cations and anions using non-selective anion- and cation-exchange membranes, which corresponds well with the previous investigation. A high global water recovery (95%) of the WWTP system was achieved from the integrated RO-ED system. The economical analysis shows that to desalinate 1m^3 of the RO concentrate, the cost is around 0.66 Euro, resulting in a extra cost for the RO-filtrate of 0.22 Euro. Thus, in this case, ED is both technically and economically proved to be a good option to treat RO concentrates.

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